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Fabricating multifunctional silver nanoparticles-coated cotton fabric

Mohammad Shateri-Khalilabad *, **Mohammad E. Yazdanshenas, Ali Etemadifar**

Department of Textile Engineering, Yazd Branch, Islamic Azad University, Yazd, Iran

Abstract   Silver nanoparticles with high density coating were formed on the surface of cotton fabric through pre-activation by potassium hydroxide and in situ reduction of silver nitrate. The silver nanoparticles-coated fabric was then reacted with hexadecyltrimethoxysilane, which resulted in the formation of low surface energy layer around the fabric. The fabrics were characterized by reflectance spectrophotometry, scanning electron microscopy, energy dispersive X-ray spectroscopy and X-ray diffraction patterns. Wettability properties were measured by contact angle goniometry and shedding angle technique. Antibacterial activity was determined against Gram-positive *Staphylococcus aureus* and Gram-negative *Escherichia coli*. Ultraviolet-blocking was measured via transmittance data in the range of 280–400 nm. Microscopy images showed the formation of uniform and high density coating of silver on the surface of cotton fibers. The fabric showed hydrophobicity with CA of 158 ± 4.3° and SHA of 7°. Also, it demonstrated a high antibacterial activity against two bacterial challenges, as shown via formation of inhibition zone. Excellent ultraviolet protection of the fabric was demonstrated by ultraviolet protection factor value of 296. The fabric also showed good durability against repeated laundering.

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1. Introduction

Wetting behavior of a solid surface is controlled by geometric structure as well as chemical composition of the material. Generally, for the conversion of a hydrophilic surface into a super-

hydrophobic surface, two key parameters are essential: creation of hierarchical surface structures (micro/nano-scale roughness) and low surface energy layer (Yan et al., 2011; Ma and Hill, 2006; Li et al., 2007). In such surfaces, microscopic air pockets are trapped beneath the water drops leading to a reduced contact area between the surface and liquid drops and help to create a composite interface which leads to increased macroscopic contact angle (CA) and reduced CA hysteresis and enable dirt particles to be picked up by the liquid and carried away as the liquid droplet rolls off the surface.

Inspired by the special surface structure of lotus leaves, fabrication of superhydrophobic textiles has attracted considerable interest (Zhao et al., 2010; Shateri Khalil-Abad and Yazdanshenas, 2010; Xu et al., 2012; Bae et al., 2009; Shirgho-

* Corresponding author. Tel.: +98 913 70757347; fax: +98 21 88741058.
E-mail address: m.shaterikha@iauyazd.ac.ir (M. Shateri-Khalilabad).
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lami et al., 2011; Wang et al., 2011; Shirgholami et al., 2013; Zhu et al., 2011). The only purpose of most of researches is the construction of superhydrophobic surfaces onto the textiles whereas fabrication of superhydrophobic textiles with multifunctional properties which can broaden the practical areas of the final product is rare.

Recently, scope of wettability research has been expanded to the fabrication of superhydrophobic surfaces with other functionalities to be used in advanced materials (Zhang et al., 2012; Privett et al., 2011; Shateri-Khalilabad and Yazdanshenas, 2013; Wong et al., 2011). In this regard, some superhydrophobic textiles with multifunctional properties have been prepared using new methodologies (Bayer et al., 2011; Liu et al., 2008; Simoncic et al., 2012). For example, an efficient method for introducing multifunctional properties to cellulose fibers was reported by Bayer et al. (2011). Cellulose fibers were functionalized with different ethyl-cyanoacrylate nanocomposite shells comprising submicrometer wax or polytetrafluoroethylene particles for superhydophobicity, $MnFe_2O_4$ nanoparticles for magnetic activity, CdSe/ZnS quantum dots for light emission and silver nanoparticles (AgNPs) for antibacterial activity. Simoncic et al. reported a method for the fabrication of multifunctional fabric with water/oil repellent and antibacterial properties. The cotton fabric was treated with two-component water- and oil-repellent antibacterial coatings consisting of commercial aqueous organic–inorganic hybrid precursors fluoroalkyl-functional siloxane and 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride.

Hydrophobicity, antibacterial and ultraviolet (UV) protections are three functional finishes of textiles that are highly interesting due to their high potential for practical and industrial applications. In this paper, multifunctional cotton fabric was prepared through a simple method based on coating of fabric with AgNPs and low surface energy silane. The prepared fabric gained functionalities such as water repellency, antibacterial activity and UV-blocking simultaneously.

2. Materials and methods

2.1. Materials

Silver nitrate ($AgNO_3$), ascorbic acid ($C_6H_6O_8$), potassium hydroxide (KOH) and ethanol were purchased from Merck. Technical grade ($\geqslant$85%) n-hexadecyltrimethoxysilane (HDTMS) was obtained from Sigma–Aldrich. Desized, scoured, and bleached plain woven cotton fabric was used as substrate. Before being used, the fabric was washed in warm water using a nonionic detergent to ensure removal of residual chemicals. After washing, the fabric was rinsed with warm water several times and then with cold water and finally dried in an oven. The water used in all the experiments was purified using a Milli-Q system with a resistivity of higher than $18.2\ M\Omega\ cm^{-1}$.

2.2. Formation of AgNPs on cotton fabric

The procedure for the synthesis of AgNPs involves two processing steps. In the first step, in order to activate cotton fabric, a piece (one gram) of cotton fabric was immersed in 30 ml aqueous solution of 6 M KOH and left there for

5 min at room temperature. The fabric was then removed from the solution and rinsed with distilled water several times; it was kept in wet state until next treatment. In the second step, the treated fabric was immersed in the 0.02 M $AgNO_3$ aqueous solution and agitated for 30 min. Then, it was removed from the solution and rinsed thoroughly with water. To form AgNPs, the fabric was transformed into a 0.01 M aqueous solution of ascorbic acid and stirred for 30 min. At the end of reaction, the fabric was removed from the vial and rinsed under flowing water. Finally, it was dried in an oven at 65 °C for 60–90 min. The obtained fabric was called Cotton-Ag.

2.3. Coating with low surface energy layer

Pristine and Cotton-Ag samples were immersed in the alcoholic solution of prehydrolyzed HDTMS (1 vol.%) and reacted at room temperature for 60 min. Subsequently, the fabrics were flushed with ethanol and dried in air. At the end, they were oven cured at 110 °C for 60 min. The samples, after hydrophobization, were marked with "HDTMS" after each sample, namely, Cotton-HDTMS for the pristine fabric and Cotton-Ag-HDTMS for the AgNPs-coated fabric.

2.4. Characterization

The reflectance spectra of the samples were recorded on a Lambda 35 UV–Vis spectrophotometer (Perkin Elmer Instruments Co. Ltd., USA). Color coordinates were determined by color measurement software using the three coordinates (L^*, a^*, and b^*) of CIELAB color system at D65/10°. Surface morphology of the fabrics was examined by a KYKY-EM3200 scanning electron microscope after sputter coating with a very thin layer of gold. To determine the elemental composition of the fabric surface, an energy-dispersive X-ray (EDS) detector was used with the VEGA TS 5136XM SEM. EDS spectra of the samples were recorded without gold coating. X-ray diffraction (XRD) patterns of the samples were recorded on a Philips PW3040 X-ray diffractometer system by monitoring the diffraction angle from 5° to 80° (2θ) using monochromatized Cu Kα (k = 1.54051 Å) radiation. Silver content of the fabric was measured by ICP OES (Varian Vista Pro, argon plasma, Ag 328.068 nm excitation, Ag sensitivity 0.004 mg L^{-1}, Australia) according to the previous work (Shateri Khalil-Abad et al., 2009). Static water contact angle (CA) measurements were completed on a self-developed goniometer apparatus coupled to a high resolution camera. Distilled water droplets with the volume of 5 μL were carefully dispensed from a syringe onto the fabric surfaces. The water CA was measured 60 s after the droplets were placed on the fabrics' surface. For statistics, 4 separate measurements were performed in different locations and the results were averaged to obtain mean and standard deviation. Water shedding angle (SHA) measurements were determined according to the work by Zimmermann et al. (2009). The water SHA was determined for 10 μL water droplets and 2 cm needle-substrate distance. The test was performed 5 times to indicate SHA. UV-blocking properties were evaluated according to the AATCC test method 183-2004 (transmittance or blocking of erythemally weighted ultraviolet radiation through fabrics) with a Perkin-Elmer Lambda 35 UV–Vis spectrophotometer, equipped with an integrating

sphere. Washing durability was investigated according to the ISO 105-C10:2006 (C) with 5 g L^{-1} solution of Trition X-100 nonionic detergent, with the liquor ratio of 50:1 at 50 °C for 45 min. The samples were washed for five cycles.

Antibacterial activity of the samples was evaluated using disk diffusion method. *Escherichia coli* (*E. coli*, ATCC 25922) and *Staphylococcus aureus* (*S. aureus*, ATCC 25923) were used as model challenge of Gram-negative and Gram-positive bacteria. Sterilized nutrient agar (cooled to 47 ± 2 °C) was dispensed into each Petri dish by pouring 15 ± 2 ml. Agar was allowed to gel firmly before inoculating. Bacterial inoculums were inoculated on the surface of the sterile agar plate and then the test specimens were gently pressed to the agar surface with a sterilized spatula. All the plates were incubated at 37 ± 2 °C for 18–24 h and examined to see whether a zone of inhibition was produced around the samples.

3. Results and discussion

Superhydrophobic surfaces are usually made by proper combination of hierarchical (micro/nanoscale) surface structure and low surface energy coatings. Cotton fabric is a rough, porous substrate produced by interlacing threads (composed of micrometer-sized cellulose fibers) placed perpendicular to each other. Consequently, microscale roughness is naturally provided and further nano-scale roughness is necessary for the formation of hierarchical structure to become superhydrophobic. Here, cotton fabric with a hierarchical structure was prepared by in situ synthesis of AgNPs on cotton cellulose fibers. Before fabric coating, activation of the cotton surface was performed through alkali treatment, as the main key for the synthesis of high density coating of AgNPs. Particle size, morphology and number of adsorbed AgNPs were found to

be affected by type and concentration of alkali in the pre-activation solution (Yazdanshenas and Shateri-Khalilabad, 2012a,b). It was found that, using KOH, more AgNPs could be formed on the fabric surface. Therefore, KOH was selected to activate cotton surface. The high concentration of KOH is necessary for the formation of AgNPs with high surface coverage. As the sample was introduced into the KOH solution, the surface hydroxyl groups of cellulose fibers were hydrolyzed and converted into the potassium salt of Cell.O$^-$K$^+$. In the next step, the potassium salt of Cell.O$^-$K$^+$ was exchanged with the silver salt of Cell.O$^-$Ag$^+$ in the aqueous solution of AgNO$_3$. Subsequently, in situ formation of AgNPs on fabrics was carried out via reduction of Cell.O$^-$Ag$^+$ by its treatment with ascorbic acid solution. Through this process, hierarchical cotton fabric was prepared.

For hydrophobicity modification, the prepared fabric was then reacted with HDTMS, which caused formation of a low surface energy layer around the fibers. HDTMS attached to cotton cellulose by naturally occurring hydroxyl groups. HDTMS was hydrolyzed to form a reactive silanol group that reacted with other silanol groups to form chains of siloxanes. The process was as follows: (1) HDTMS hydrolysable groups were hydrolyzed; (2) molecule was condensed to form oligomers; (3) oligomers were activated and silanols were formed; (4) silanol bonds were made with each other and with hydroxyl groups on cotton cellulose through condensation reactions.

3.1. Reflectance spectra and color coordinates

Coating of fabric with AgNPs was demonstrated via assessing color change of the fabric (Dubas et al., 2006; Kelly and Johnston, 2011) and measuring reflectance spectra at wavelengths of 400–700 nm. Reflectance spectra of the fabrics are
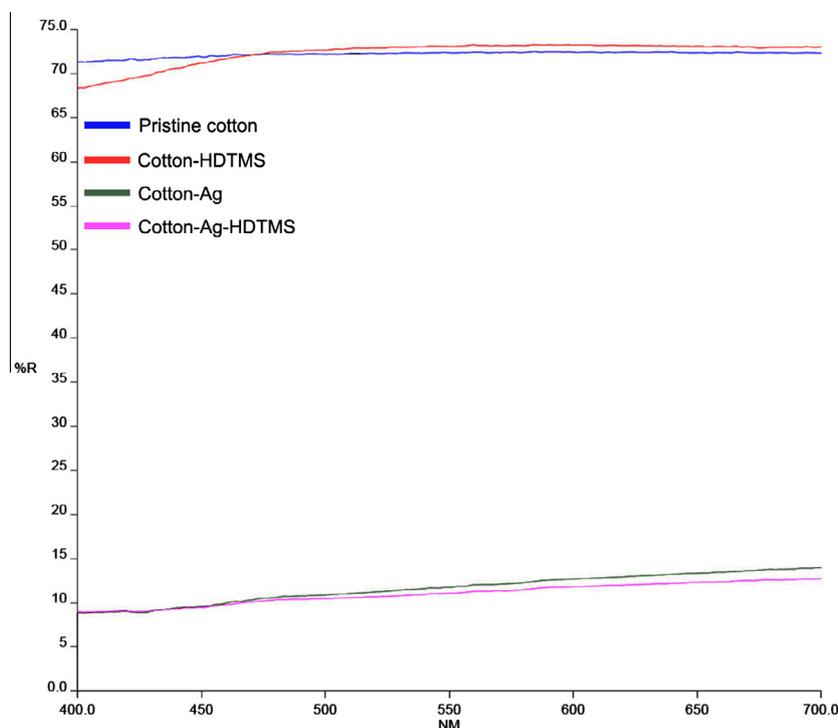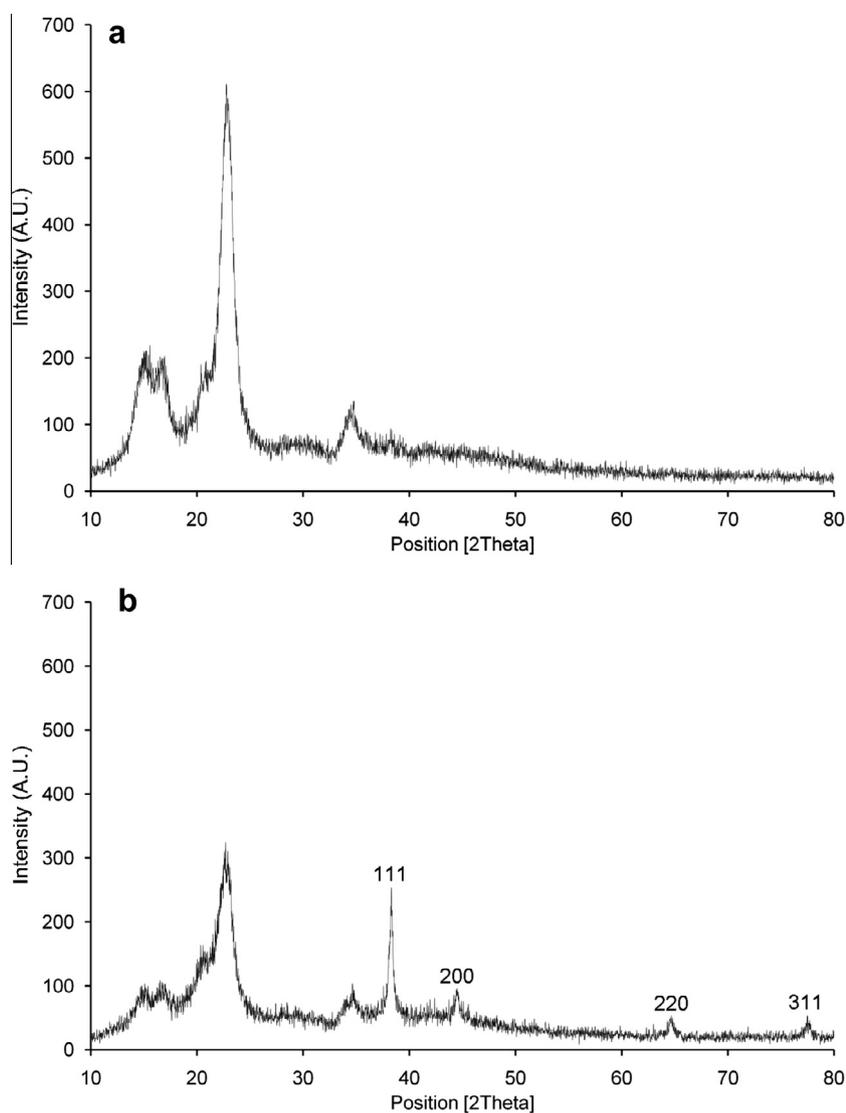

Figure 1   Reflectance spectra of the pristine and Cotton-Ag samples.

Table 1 Color coordinates (CIE L^* a^* b^*) and color differences (ΔE) of the fabrics.

Fabric	L^*	a^*	b^*	ΔE
Pristine	88.13	−0.17	0.39	–
Cotton-HDTMS	88.43	−0.58	1.57	0.63
Cotton-Ag	40.84	0.99	5.96	47.63
Cotton-Ag-HDTMS	39.75	0.81	4.48	48.56

shown in Fig. 1. The pristine cotton fabric changed its color from white to dark green after treating with AgNO$_3$, which indicated the formation of AgNPs on the fabric. The broad nature of absorption band of Cotton-Ag sample indicated that the sample contained both aggregated particles and a range of particle sizes (Kelly and Johnston, 2011). The CIE L^*, a^* and b^* values, quantitatively characterizing the observed color of the samples, are shown in Table 1. The results showed visible color difference between pristine and Cotton-Ag samples with
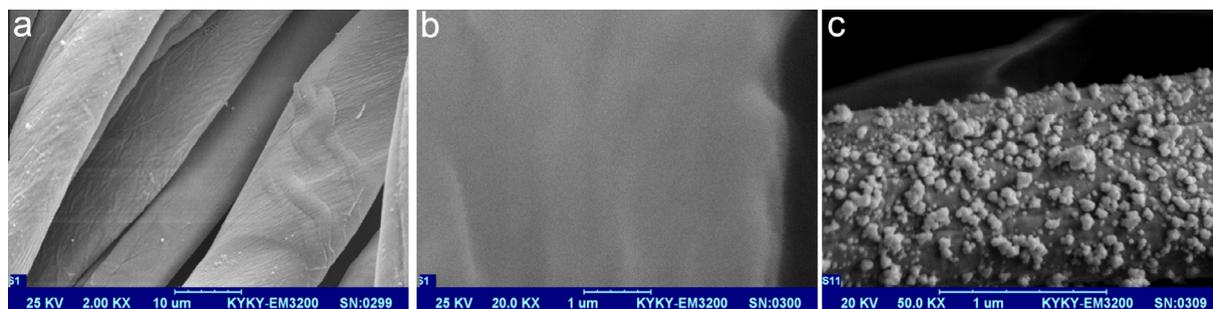
Figure 2   SEM micrographs of the (a) pristine and (c) Cotton-Ag samples (b) higher magnification of (a).
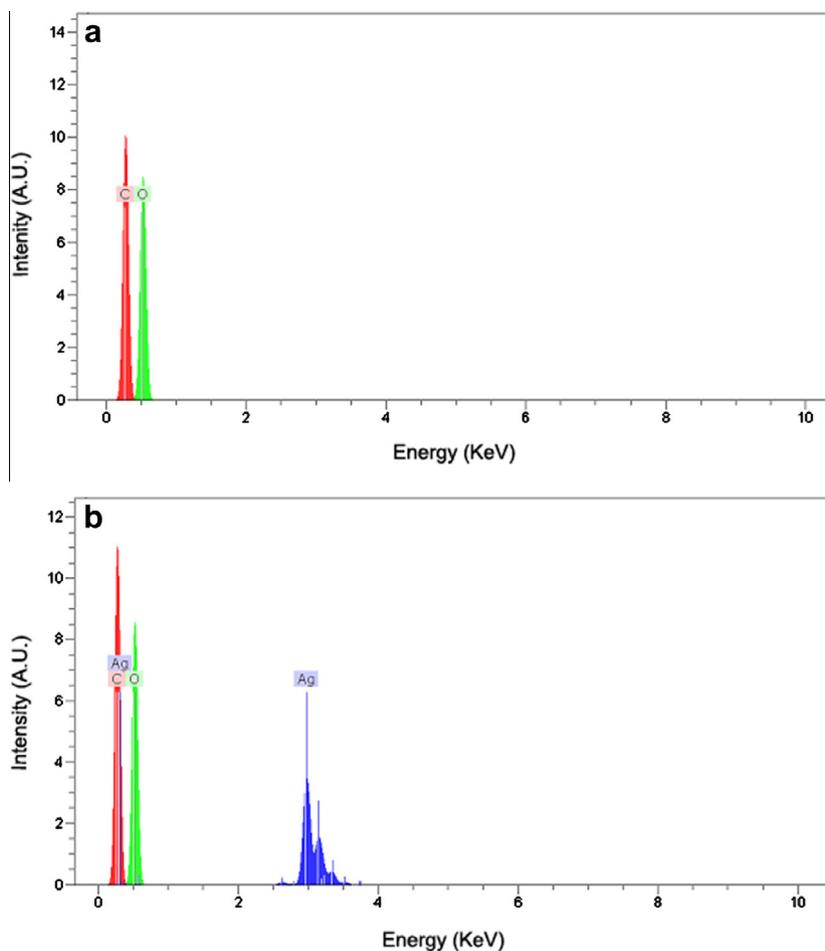
Figure 3   EDS spectra of the (a) pristine and (b) Cotton-Ag samples.

Figure 4   X-ray patterns of the (a) pristine and (b) Cotton-Ag samples.

ΔE of 47.63. In addition, spectra of the samples after coating with HDTMS were recorded. After HDTMS coating, a small difference was observed in reflectance spectra and color coordinates, which demonstrated transparency of HDTMS coating in comparison to the uncoated ones. For reducing the undesirable brown color on fabrics, hybrids of Ag-TiO$_2$ or Ag-SiO$_2$ nanoparticles can be used, which was not the focus of this work.

3.2. SEM images

Surface morphology of the samples was investigated by electron microscopy. The SEM micrographs are shown in Fig. 2. Images of the pristine fabric (Fig. 2a and b) show smooth longitudinal fibril structure of the fibers without any contaminating particles on their surfaces. However, AgNPs with high surface coverage were formed on the AgNO$_3$ treated fabric. Some of the AgNPs were agglomerated and caused the formation of micro-particles. The small AgNPs were adsorbed on both uncovered fibers and larger aggregated particles

(Fig. 2c). The images demonstrated that a rough surface with a hierarchical structure was formed by in situ deposition of AgNPs on cotton fabric.

3.3. EDS spectra

Surface chemical elements of the fabrics were determined by EDS spectroscopy. Fig. 3 shows EDS spectra of the pristine and Cotton-Ag fabrics. Peaks of C and O were detected on the pristine sample (Fig. 3a). However, for the Cotton-Ag sample, a new peak appeared at ca. 3 keV which, could be attributed to the silver signal of AgNPs (Fig. 3b). A strong peak of Au at ca. 2 keV was related to gold coating of the fabrics.

3.4. XRD patterns

XRD patterns of the pristine and Cotton-Ag samples are shown in Fig. 4. Compared to the pristine fabric, four new peaks at 2θ values of 38.1°, 44.3°, 64.5° and 77.5° were detected, which were respectively attributed to the diffraction
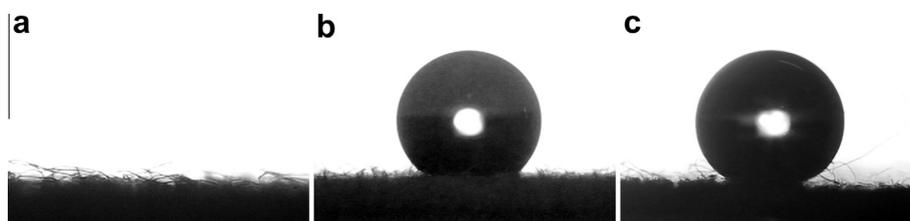
Figure 5 Optical micrographs of 5 μL water droplets on the surface of the (a) pristine, (b) Cotton-HDTMS and (c) Cotton-Ag-HDTMS samples.

peaks of the (111), (200), (220) and (311) planes of silver with cubic structure reported in the International Center for Diffraction Data (JCPDS data number 04-0783 card) on Cotton-Ag sample. No characteristic peaks were observed for other impurities such as AgO. The mean crystal size of AgNPs was estimated as 53 nm by analyzing the broadening of the (111) reflection calculated by the Scherrer equation (Waseda et al., 2011). The inconsistent result of particle size between SEM images and XRD might be due to the difference between coherent length and particle size. It may be also due to deconvolution of the instrumentation broadening.

3.5. Hydrophobicity properties

As demonstrated in the SEM images, coating of the fabric with AgNPs caused formation of hierarchical structure, i.e., AgNPs on the surface of cellulose microfibers. In order to create a hydrophobic surface, the fabric was further modified by self-assembly of HDTMS to reduce surface energy.

Wetting properties of the fabrics were analyzed by measuring water droplet CA and SHA (Fig. 5). As shown in Fig. 5a, the pristine fabric was completely wetted by water through rapid spreading of water droplet due to the presence of abundant hydroxyl groups on the surface of cotton cellulose. Cotton-Ag sample also showed the same behavior so that water droplet was gently adsorbed on its surface, demonstrating that coating with AgNPs did not change wetting properties of the fabric. Fig. 5b shows CA of 5 μL water droplet placed on the Cotton-HDTMS sample. For this sample, CA and SHA were 135.5 ± 5.2° and 31°, respectively, which demonstrated its hydrophobicity properties. However, for the AgNPs-coated fabric (Cotton-Ag-HDTMS), CA increased and SHA decreased. Superhydrophobicity properties with CA of 158 ± 4.3° (Fig. 5c) and SHA of 7° were observed. The increased hydrophobicity can be attributed to the increase in surface roughness of the fabric as results of its coating with AgNPs. In this sample, hydrophobicity was stable and did not reduce over time. In conclusion, a superhydrophobic surface with high CA and small SHA was prepared, ascribing to two-level surface structure of the fabric.

3.6. Antibacterial properties

Antibacterial properties were evaluated against Gram-negative E. coli and Gram-positive S. aureus bacteria using agar diffusion technique. Fabric samples were placed on bacteria-inoculated agar plates and were visualized for antibacterial activity (Fig. 6). The pristine fabric was used as a control. The control samples did not show any antibacterial activity (high bacterial

growth as indicated by bacterial growth lawn, large indistinguishable collection of colonies). Similarly, bacterial growth was observed for the hydrophobized sample (Cotton-HDTMS), which demonstrated inability of HDTMS to eliminate bacteria. Conversely, a distinct and significant inhibition zone was observed around the Cotton-Ag and Cotton-Ag-HDTMS samples placed in contact with both E. coli and S. aureus. The formation of HDTMS layer on the surface of Ag-coated fabric did not decrease the antibacterial effect. The reason for this phenomenon is not clear to us. However, it may be due to uncompleted or very low thickness of low surface energy coating.

The formation of inhibition zone clearly indicates that the mechanism of biocidal action of the fabric is due to the leached Ag^+ ions. The higher diameters of inhibition zone around the samples placed on agar inoculated with S. aureus show that the fabrics have a better antibacterial effect against S. aureus. Such results are in a good agreement with the previously reported literature (Maneerung et al., 2007).

3.7. UV-blocking

Fig. 7 shows transmittance spectra of the fabrics in the range of 280–400 nm. Spectrum of the pristine fabric showed that high percent of the UV-A and UV-B radiation could penetrate in the fabric and UV-blocking ability on the UV-B region was higher than UV-A region. Transmittance spectrum of the Cotton-HDTMS sample demonstrated increased UV-blocking ability, compared to the pristine one. Transmittance spectrum of the Cotton-Ag sample indicated that it can block high percent of the UV-A and almost all of the UV-B radiation. As compared to this sample, spectrum of the Cotton-Ag-HDTMS
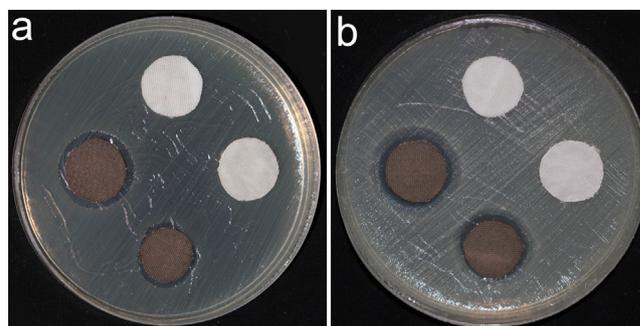
Figure 6 Antibacterial activity of the fabrics placed on the agar plate inoculated with (a) E. coli and (b) S. aureus. (top) Pristine fabric (right), Cotton-HDTMS, (left) Cotton-Ag and (bottom) Cotton-Ag-HDTMS.
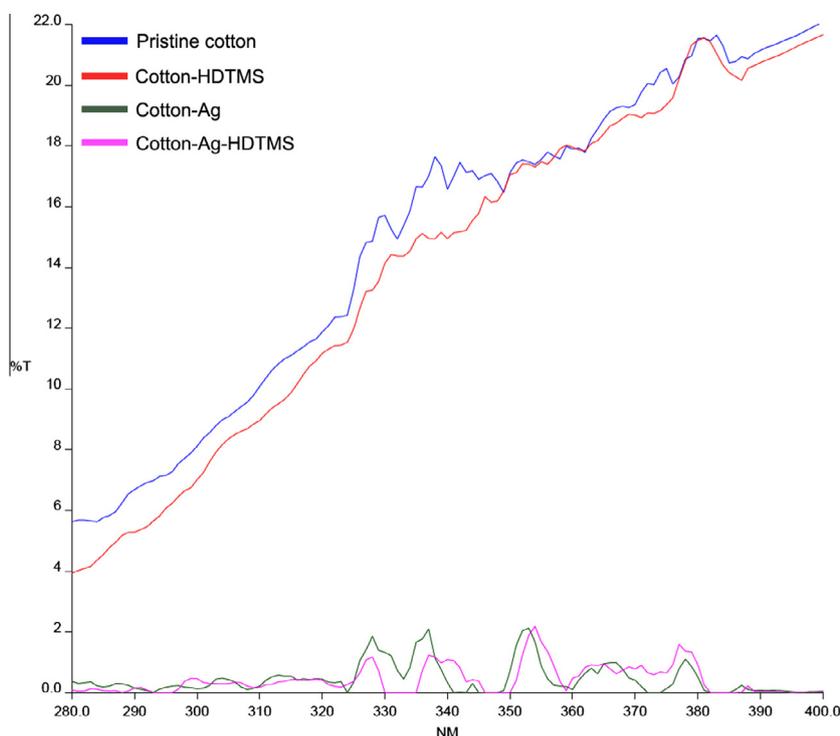
Figure 7 UV-transmittance spectra of the samples.

Table 2 UV-blocking properties of the fabrics.

Sample	Percentage of transmittances (%)			UPF value
	UV-R 280–400 nm	UV-A 315–400 nm	UV-B 280–315 nm	
Pristine	14.96	17.87	7.91	9.4
Cotton-HDTMS	14.11	17.14	6.74	10.4
Cotton-Ag	0.44	0.52	0.26	270
Cotton-Ag-HDTMS	0.42	0.52	0.19	296

sample showed a small increase in its blocking ability. Actually, spectra of Cotton-Ag and Cotton-Ag-HDTMS samples were largely close to each other and there was only a small difference between them. Since the actual damage to human skin from UV radiation is a function of wavelength of the incident radiation, with most of the damage done by radiation in the range of 300 to 320 nm, fabrics must demonstrate effectiveness in this range (Schindler and Hauser, 2004). The results revealed that the obtained superhydrophobic fabric can provide excellent UV-blocking in the mentioned range and also in the UV-A range.

The UPF rating indicates how effective a fabric is at blocking out solar ultraviolet radiation and, the higher the UPF value, the better the protection of garment would be. UPF is defined as the ratio of average effective irradiance calculated for skin to average UV irradiance calculated for skin protected by the fabric. UPF value of the fabrics is calculated using mean percentage transmission in the UVA (315–400 nm) and UVB (280–315 nm) regions. Typically, fabrics with UPF value of more than 40 are considered as providing excellent protection against UV radiation [34]. The results of UPF values and

transmittance of light in the ranges of UV (UV-B, UV-A, and UV-R) for the fabrics are presented in Table 2. These results showed that the pristine fabric with UPF value of 9.4 provided poor protection against UV radiation. The UPF value of less than 15 had no protection against transmittance of UV radiation through fabric and onto skin (AS/NZS 4399, 1996). The coating of fabric with HDTMS improved its UV-protection. The UPF value for the Cotton-HDTMS sample increased to 10.4, which indicated effectiveness of the HDTMS coating for protection against UV radiation. For the Cotton-Ag, UV transmittance decreased significantly (percentage of transmittance in the UV-B was lower than UV-A) and UPF value increased to 270 showing its excellent ability to block UV radiation. UPF value of the Cotton-Ag-HDTMS sample had increased more and reached 296 as a result of HDTMS coating, which demonstrated its outstanding UV-blocking ability. High UV absorption as a result of dark brown color of the AgNPs-coated fabric could be a reason for such high UV-blocking (Gorenšek and Recelj, 2007).

3.8. Washing durability

To evaluate washing durability, the Cotton-Ag-HDTMS sample was washed 5 cycles and then its hydrophobicity, antibacterial and UV-blocking properties were investigated. After washing, CA of the sample reduced to 149 ± 5.2° and SHA increased to 13°, which showed a small reduction in hydrophobicity. The clear inhibition zones were also observed around the washed sample against both Gram-negative and Gram-positive bacteria, which demonstrated its antibacterial activity. Also, minor reduction of UV-blocking ability with UPF value of 261 was observed. The results from these experiments demonstrated good washing durability of the prepared

fabric. Good washing durability of the finished product could be related to in situ formation of AgNPs on fabric (Yazdanshenas and Shateri-Khalilabad, 2012a,b) and their coating with HDTMS, which could protect them from being detached during washing.

4. Conclusions

Cotton fabric with multifunctional properties was prepared through coating with AgNPs and low surface energy layer. The fabric showed superhydrophobicity with CA of higher than 150° and SHA of lower than 10° as a result of forming a hierarchical structure. Antibacterial activity of the fabric against both Gram-positive and Gram-negative bacteria was demonstrated by observing inhibition zone. In addition, the fabric showed excellent UV-blocking with a UPF value of 296. It is expected from the functionalized fabric to have a high potential for the preparation of advanced multifunctional textiles, which could revolutionize their end uses.

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